ERRATA

NIST SPECIAL PUBLICATION 801 dated October 1990
Laser Induced Damage in Optical Materials: 1989

The following pages were inadvertently omitted and are attached: page 24a, page 38a, page 441a, and page 508a.

The "comments" sections of the following papers were collated incorrectly; for example, the first paper (abstract only) listed below is on page 9 but the "comments" (questions and answers) are on page 323. The second paper, listed below, starts on page 10 and ends on page 24; however, the comments on page 24 do not pertain to the second paper. Comments pertaining to the second paper were not printed and thus are attached as page 24a.

COMMENTS

Question: Does surface absorption affect your measurements?

Answer: Yes it does. The effect appears in the extinction coefficient or k value but not in n, the real part of the index of refraction.

COMMENTS

Question: Have you or do you have any plans to apply this technique to dielectric coatings on substrates?

Answer: No, Sir.

Question: There is a fair amount of activity in this area in Europe, there is a group of people out there that feel that this can be applicable for example to adhesion testing as well as hardness correlated with damage.

Answer: I am unaware of this activity.

Question: Has a relation between subsurface damage and laser induced damage threshold been shown to your knowledge? I have not kept up with this since House and co-workers at the Air Force Weapons Lab were not able to show that dependence so I was just curious.

Answer: If we go to the work of Wood, and the work of Hurt, the answer to your question would be yes. Whether more than that has been done I am unaware.

Question: Wondering if how far you can or how much information you can get about the depth and the difference in hardness of the surface layer by analyzing the slopes on your curves.

Answer: What you are talking about is utilizing the exponent, and the answer to your question is that you can learn quite a bit.

Question: I just might point out a possible difference in interpretation of indention measurements. Some work that Martin Stickley sponsored at Battelle Northwest Laboratory on sputtered deposited metal films. We performed laser damage testing and microindentation testing on these surfaces and they gave similar results. The microindentation did work harden surfaces although those films had nothing like the microscopic properties that you would see in a work hardened surface. I suggest that there is perhaps ambiguity in those measurements.

Answer: True, and that is one of the reasons why we resort to a variety of auxiliary techniques in trying to address these problems. Typically we would use these x-rays to tell us if we had not had a conversion from a possibly glassy material to a crystal material. That is one thing. I see quit a few things that have been plated or sputtered typically start out as amorphous material. With modest heating they convert with all sort of disruptions - we do a lot of metallography, we do some transmission microscopy now, but your point is well taken.

Question: What's the size of the sphere you are using?

Answer: I am using a 1.6 millimeter ball; at the most I can apply 10 N, typically I am interested in the first micron which means I put down a footprint diameter of the ball - on the surface it is about 80 microns.

Question: You are aware there is a nanosphere indentation technique with a much higher resolution to resolve the nonuniformity of the hardness on the surface.

Answer: I would ask you not to think in terms of hardness because hardness is really a measurement that's made on a residue that's lost elastic effect, it's lost the inelastic effect, it reflects what people call elasticity, so in answer to your question, I am not aware of the microball, I am aware of the nanoindenter, which uses the Burkowits indenter, which is a three-sided pyramid, which philosophically is totally different than what I am describing this morning. I suggest that when you try to measure surface properties you like to engage as much as the surface at the same time going in as little as possible, and so my preference will be to go to a 4 or 5 mm ball. We are trying to do that and at the same time develop diamond 25 micron balls for some fracture work we are doing.

Question: Could you explain the relationship of this work to Norm Boling's presentation at this meeting on microwaves?

Answer: Yes. There are a number of different processes that use plasma CVD. Most of them are low temperature. Norm Boling has a process that runs at a much lower temperature than this. We feel that it is necessary (as a matter of fact, we've had this discussion with Norm) that you have to have the high temperature. We see that in the fused silica material that we buy. And, sure enough, we have tested coatings that have been made by the low temperature process and they're not anywhere as good as the E-beam coatings that we have. So, the key really is a combination of temperature here and the plasma to initiate the reaction. You can drop the temperature a little bit by using a plasma. Most of the chemical deposition processes for fiber optics, like Corning uses, run at well over 1500°C, perhaps 1300°C for some of the reforms. But that's a key. When you think about it, one of the reasons for this result is that thermodynamically and kinetically you start to get to the point where things are favorable. If you get a piece of crud into the system, the chance of being able to oxidize it under those conditions becomes quite high. So, in a way, you also have a way to get rid of contaminants if they end up in there.

Question: What is the spectral bandwidth to the 1/2 point?

Answer: It is narrow, but it's about twice as wide as what you'd predict. And that's really a drift in the process, particularly the mass flow controllers when they lay the material down or they introduce the material.

Question: Have you characterized the water content in these films?

Answer: Yes. In fact, these are pretty dry. We were worried that water might cause a lower damage threshold, but we actually tested fused silica tubes that had a range in water concentration from about 10 to 110 parts per million. The highest one was the one that had the highest water in it. We don't know why that is, but I don't think the water will bother us.

Question: Are there any fundamental limitations using SiO_2 as a carrier material. Can you get a larger index contrast, basically?

Answer: Part of the problem is, we tried to go with larger index contrast, but you start to introduce a fair amount of stress by some of the dopants. So, as a consequence, it will damage at a lower threshold. For example, we tried to introduce higher germania doping concentrations for that reason. In fact, we went all the way to pure germania, cycling from SiO_2, and the stresses are to high to give you high damage threshold. So, unfortunately, I do believe it's a requirement that you have to stay in that region. Somebody might do some research and find out some better dopements; we would hope for that in the future. But right now, it's not that way.

COMMENTS

Question: Have you done any thermal cycling at all? Have you temperature cycled the temperatures up or down?

Answer: No, we have not.

Question: You used a zirconium chloride as a binder. Was there any residual chloride in your films?

Answer: We didn't do any chemical analysis on that, but I suspect that there are probably some chloride residues in this coating. If you really wanted to eliminate this chlorine you would have to heat it quite high. The process is working for what we want to do, so maybe it's not necessary to heat these substrates. It would be difficult especially if you want to heat a large mirror.

Comment: I just want to make an observation. It's very interesting at this meeting that laser annealing seem to be very popular and is discussed as newly discovered. I recall that ten years ago this process was well described by experiments with multi-pulse excimers in reports by Fulton at Los Alamos. The technique now seems to be spreading. It is gratifying that it is again finding good use.

Question: Is spin application preferable to dip or other forms of application?

Answer: If I had to choose the process to use to coat I would use the dip coating technique. With it, if you have one piece if dust in the atmosphere during coating operation you get one local defect. In contrast, with the spin coating technique if you had the same dust you will have turbulences during the spinning of the substrate so you will give some energy to the dust and you will have a very bad defect on your coating. We are obliged to use the spin coating because we can't prepare a large amount of coating solution. That's the main reason why we use it now.

Question: Have you looked at other refractory oxide candidates such as samarium oxide for possible application in this area?

Answer: Yes, we worked first with titania. We also tried yttria. The problem is that this material ia very expensive. You have to consider in this technology to try to lower the price as much as possible. For the Athena project, we are speaking about many optical components and we try to keep the cost down. With yttrium or other materials we spend more money.